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(54) METHOD OF CLEANING FOOD AND BEVERAGE MANUFACTURING AND HANDLING EQUIPMENT

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None

See application file for complete search history.

(56) References Cited

U.S. PATENT DOCUMENTS

6,986,817	B2	1/2006	Riefenstein et al.	
8,685,173	B2 *	4/2014	Ahmed	C11D 3/30
				134/22.1
2003/0015219	A 1	1/2003	Kravitz et al.	
2003/0101939	A 1	6/2003	Van Den Berg	
2005/0090542	A 1	4/2005	Aronhime et al.	
2006/0035808	A1	2/2006	Ahmed et al.	

FOREIGN PATENT DOCUMENTS

CA	2143195	6/2002
DE	19921709	12/1999
EP	0784930	7/1997
FR	2602955	2/1988
KR	100311580	12/2001

OTHER PUBLICATIONS

The International Search Report and Written Opinion dated Dec. 23, 2009, in the corresponding PCT/US2009/050828 filed Jul. 16, 2009. The Summons to Attend Oral Proceedings Pursuant to Rule 115(1) EPC dated Oct. 14, 2013, in European Application No. 09 798744.0. The Patent Examination Report No. 1 dated Jul. 16, 2013, in Australian Application No. 2009270819 filed Jul. 17, 2008.

The European Search Report dated May 30, 2012 in the European Application No. 09 798744.0.

The Office Action issued on Aug. 6, 2013, in Japanese Patent Application No. 2011-518907.

The Office Action issued on Jun. 15, 2011, in New Zealand Patent Application No. 591184.

* cited by examiner

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(57) ABSTRACT

The present invention is generally directed toward methods of cleaning and descaling surfaces contaminated with food soils, especially clean-in-place systems. More particularly, the methods according to the present invention also provide for sanitizing of surfaces contaminated with food soils. Thus, there is provided a single cleaning cycle that may clean, sanitize, and descale food-soiled surfaces, and in certain embodiments, without the need for a pre-rinse step, using a non-chlorine detergent composition. The methods comprise a cleaning step and a post-rinsing step. In certain embodiments, a pre-rinse step is utilized, in which the volume of pre-rinse fluid used is less than 50% of the volume of cleaning solution used in the cleaning step.

17 Claims, No Drawings

METHOD OF CLEANING FOOD AND BEVERAGE MANUFACTURING AND HANDLING EQUIPMENT

RELATED APPLICATION

The present application is a continuation of U.S. patent application Ser. No. 13/054,004 filed Mar. 29, 2011, entitled METHOD OF CLEANING FOOD AND BEVERAGE MANUFACTURING AND HANDLING EQUIPMENT, 10 which is a national stage submission under 35 U.S.C. 371 of International Patent Application No. PCT/US2009/50828, filed Jul. 16, 2009 and issued as U.S. Pat. No. 8,685,173 on Apr. 1, 2014, which claims the benefit of the U.S. Provisional Patent Application No. 61/081,634, filed Jul. 17, 15 2008. All of the foregoing applications are hereby incorporated by reference in their entireties.

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention is generally directed toward methods of cleaning and descaling surfaces of equipment contaminated with food or beverage soils. In addition, the methods of the present invention generally result in a 25 substantial reduction of bacteria and/or other microorganisms present on the surfaces of the equipment. More particularly, the methods according to the present invention comprise a single cycle cleaning method that can reduce water usage or entirely obviate the need for a pre-rinse step, 30 and is especially suited for use with clean-in-place systems.

Description of the Prior Art

Clean-in-place (CIP) systems are commonly used in many food industries, including dairy, beverage, brewing, and processed foodstuffs. These systems are also commonly 35 used in the pharmaceutical and cosmetics industries. These systems are designed such that the interior pipes, vessels, process equipment, and associated fittings can be cleaned without disassembly of the equipment. Adequate cleaning of food preparation surfaces is a necessity to ensure the safety 40 of the food supplied to consumers. This is especially true for the dairy industry, food preparation and processing plants, including food and beverage plants, and particularly in the area of milk handling and storing. Fresh milk must be immediately cooled and refrigerated after being obtained 45 from the cow in order to prevent the milk from spoiling. Consequently, the piping systems, equipment, storage tanks, and utensil surfaces which handle the flow of milk must be cleaned after each milking in order to remove milk soils so as to prevent contamination of the fresh milk supply during 50 subsequent milking operations. Most dairies operate using at least two milkings per day. This means that the CIP systems must be cleaned at least twice per day.

Traditionally, CIP systems in North America (the United States and Canada) have always been cleaned using chlorinated alkaline or alkaline detergents and at least a three-step process. In the first step, the system is pre-rinsed with water at about 37-49° C. (100-120° F.). The goal in this step is to soften or melt the milk fats, without using water so hot as to denature the milk proteins and create scale. It has been 60 conventionally thought that this step was a necessary pre-requisite to the cleaning process and for scale inhibition. In the second step, the system is washed with hot water of no less than 49° C. (120° F.), and usually closer to about 70-80° C. (158-176° F.), using the chlorinated alkaline or alkaline 65 detergent, which is circulated through the system for about 8-10 minutes. The chlorinated alkaline detergent hydrolyses

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and dissolves milk fats, proteins, and carbohydrates; removes protein deposits and prevents film build-up. Finally, the system is post-rinsed to remove the detergent residues. Often this step involves an acid rinse at about 38-49° C. (100-120° F.) that also helps remove scale, followed by a sanitizing step, or a single acid sanitizing rinse at about 21-38° C. (70-100° F.) using a combination acid sanitizer.

In Europe, the conventional cleaning process alternates between chlorinated alkaline detergent and acid detergent. According to these processes, one type of detergent is used for the morning cleaning, while the other type of detergent is used for the evening cleaning. These detergents are often combination cleaner-sanitizers. In the typical cleaning process, the system is pre-rinsed with ambient temperature or warm water, and then washed with hot water at about 60-80° C. using the acid or chlorinated alkaline detergent. The systems is then post-rinsed with ambient temperature water.

Chlorinated cleaning detergents are effective for cleaning CIP systems. However, the use of chlorinated alkaline detergents has several drawbacks, including corrosion and degradation of polymeric gaskets, hoses, and appliances in the milk handling equipment, as well as environmental concerns from discharge of the cleaning water from the system. Furthermore, chlorine concentrations are not easy to maintain in detersive solutions. The effectiveness of chlorine on protein soil removal diminishes as solution temperature and pH decreases. Also, chlorine can react with organic materials to form carcinogenic chlorocarbons, such as chloromethane, di- and trichloromethane, and various derivatives of chloroethane.

More recently, attempts have been made to increase the efficiency of cleaning CIP systems, as well as reduce the environmental impact of such processes, save energy, and reduce water consumption. However, conventional methods are not suitable to water re-use methods that are currently being explored. In these re-use methods, the water from the earlier cycle (i.e., the rinse water or wash water) is stored and reused for either the pre-rinse or wash water in the subsequent cleaning. However, acidic water from the postrinse or the previous wash water partially neutralizes the alkalinity of the detergent used in the next cleaning cycle, or vice versa. This inhibits the effectiveness of the overall cleaning process and often results in the need for additional cleaning cycles thereby eliminating the benefits of recycling the water in the first place. Therefore, additional methods of improving the process of cleaning CIP systems have been sought.

WO 2005/090542 discloses a method of cleaning dairy equipment without the use of chlorine-containing alkaline detergents or a pre-rinse step. The method utilizes a cleaning solution containing at least one peroxide, which claims to take advantage of the lactoperoxidase enzymes and thiocyanate inherent in the dairy residue to be removed from the system. Lactoperoxidase and thiocyanate are disclosed in WO 2005/090542 as being a natural germicidals and antispoilants. The hydrogen peroxide in the disclosed cleaning solution activates the lactoperoxidase enzyme in the milk soil, which in turn kills the enzymes responsible for milk spoilage. A disadvantage to this system is that it is specific to dairy processing systems and would not work to clean other systems that do not have the lactoperoxidase enzymes or thiocyanate inherent in the soils to be removed from the dairy processing equipment. In addition, comparative testing of the disclosed peroxide cleaning solution indicates that there is only a 3-log reduction in the population of bacteria, which is not an acceptable level to be considered an antimicrobial or sanitizer in Europe or the United States.

Thus, there exists a real and substantial need in the art for a method of cleaning a clean-in-place system, which is not limited to dairy food or beverage processing plants, using a non-chlorine, acidic detergent composition capable of cleaning and descaling food preparation surfaces in a single cleaning cycle with a reduced volume pre-rinse step or entirely without a pre-rinse step, and further sanitizing under certain conditions. There is also a need for a method of recycling water from the cleaning process that avoids problems of traditional cleaning processes.

SUMMARY OF THE INVENTION

The present invention overcomes the above problems and provides cleaning and descaling functionality in a single 15 cleaning cycle with substantially decreased water usage, and often without a pre-rinse step, that is especially suited for CIP systems.

In one embodiment of the present invention there is provided a method that comprises a cleaning step in which 20 the surfaces of the soil-contaminated equipment are contacted with a cleaning solution comprising an acidic detergent composition including a fatty alkyl-1,3-diaminopropane or salt thereof having the general formula R—NH— CH₂CH₂CH₂NH₂, wherein R is a C4-C22 alkyl group. The 25 cleaning step is followed by a post-rinsing step in which the equipment surfaces are rinsed with a rinse solution thereby removing residues of the detergent composition remaining on the equipment. The above steps are performed without first performing a pre-rinsing step as is common in conventional CIP cleaning operations.

In another embodiment of the present invention there is provided a method of cleaning soiled food or beverage manufacturing and handling equipment comprising a cleaning step in which the surfaces of the equipment are contacted 35 with a volume of a cleaning solution. The cleaning solution comprises an acidic detergent composition including a fatty alkyl-1,3 diaminopropane or salt thereof having the general formula R—NH—CH₂CH₂CH₂NH₂, wherein R is a C4-C22 alkyl group. The volume of cleaning solution is 40 circulated through the equipment for a period of time to effect a reduction of the soils on the equipment surfaces. A post-rinsing step is then performed in which the equipment surfaces are rinsed with a rinse solution thereby removing residues of the detergent composition remaining on the 45 equipment. The method may also include a pre-rinse step in which a volume of pre-rinse fluid is circulated through the equipment prior to said cleaning step wherein the volume of pre-rinse fluid used is less than 50% of the volume of cleaning solution used in the cleaning step.

In yet another embodiment of the present invention there is provided a method of cleaning soiled food or beverage manufacturing and handling equipment comprising a cleaning step in which the surfaces of the equipment are contacted with a volume of a cleaning solution. The cleaning solution 55 comprises an acidic detergent composition including a fatty alkyl-1,3 diaminopropane or salt thereof having the general formula R—NH—CH₂CH₂CH₂NH₂, wherein R is a C4-C22 alkyl group. The volume of cleaning solution is circulated through the equipment in a plurality of passes, 60 portions or slugs to effect a reduction of the soils on the equipment surfaces. A first portion or slug of the cleaning solution is purged from the equipment following the first pass therethrough. After the cleaning step, a post-rinsing step is then performed in which the equipment surfaces are 65 rinsed with a rinse solution thereby removing residues of the detergent composition remaining on the equipment. The

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surfaces of the equipment have not undergone a pre-rinse step prior to the cleaning solution first pass.

In still another embodiment of the present invention there is provided a method of cleaning soiled food or beverage manufacturing and handling equipment without a pre-rinse step. The method comprises a cleaning step in which a first portion or slug of cleaning fluid (water only) is introduced into the equipment thereby contacting the surfaces thereof. The first portion of water effectively dilutions and residual 10 soil that may remain in the equipment because of inadequate draining. Subsequently, a second portion or slug of cleaning fluid is introduced into the equipment thereby contacting the surfaces thereof. The second portion of cleaning fluid comprises an acidic detergent composition including a fatty alkyl-1,3 diaminopropane or salt thereof having the general formula R—NH—CH₂CH₂CH₂NH₂, wherein R is a C4-C22 alkyl group. The first and second portions of cleaning fluid circulate through the equipment simultaneously. A post-rinsing step is performed in which the equipment surfaces are rinsed with a rinse solution thereby removing residues of the detergent composition remaining on the equipment. The surfaces of the equipment have not undergone a pre-rinse step prior to the cleaning solution first pass.

Therefore, the present invention provides an efficient method of cleaning and descaling of surfaces of a CIP system contaminated with food or beverage soils. Further, in certain embodiments, the method can also be used to sanitize soiled surfaces. Although the invention finds particular utility for CIP treatment of dairy equipment, and beverage or food processing plants, it is not so limited. Those skilled in the art will appreciate that the claimed methods can be used to clean, sanitize, and descale a wide variety of equipment, such as heat exchangers, tanks, pipes, centrifuges, evaporators, filters, extruders, coders, coolers, sieves, hydrocyclones, and ultra-, hyper-, micro-, and nanofiltration units.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

All embodiments of the present invention generally comprise both a cleaning step and a post-rinsing step. In certain embodiments, the need for any pre-rinse step is eliminated thereby saving significant quantities of water and cleaning time. However, in other embodiments, particularly those embodiments pertaining specifically to beverage handling equipment, and even more specifically to milk handling equipment, it is within the scope of the present invention to include a low-volume pre-rinse step in order to remove or flush standing beverage or milk that could not otherwise simply be drained from the equipment. As explained below, this pre-rinse step is not intended to remove excess food or beverage that is clinging to the surfaces, rather due to the design of certain CIP systems, significant quantities of free-standing beverage may remain in the system and/or system lines. Thus, in order to prevent a loss of detergent efficacy, these free-standing quantities of beverage need to be removed via a low water volume pre-rinse. Alternatively, the free-standing quantities of beverage may be diluted by circulating the cleaning solutions in two portions. The first portion of cleaning solution containing only water effectively dilutes the soil that would otherwise accumulate in the first slug of cleaning solution that circulates in the system.

Although the cleaning step may be carried out by way of a number of embodiments which are discussed in detail below, generally the cleaning step involves the contacting of a surface of the handling or processing equipment that is soiled with food or beverages with a cleaning solution at a

temperature of from about 25° C. to about 85° C., preferably from about 35° C. to about 80° C., and more preferably from about 40° C. to about 75° C., for a specified time period of from about 2 to about 20 minutes, preferably from about 6 to about 15 minutes, and more preferably from about 8 to about 12 minutes. Preferably, the surface is contacted with the cleaning solution by circulating the cleaning solution through the equipment for the specified period of time.

The embodiments of the present invention described herein are particularly suited for use with clean-in-place 10 (CIP) systems such as those found on dairy farms and in a number of food and beverage processing handling facilities. One exemplary type of CIP system comprises a batch tank in which cleaning and/or rinse solutions may be held during the cleaning cycle. The batch tank provides a container for 15 mixing the detergent into the water to be circulated through the various portions of the CIP system during the cleaning process. After completing a circuit through the equipment, the solutions are typically returned to the tank to await further circulation. Another type of CIP system foregoes the 20 batch tank and instead utilizes apparatus for adding detergent in-line as the cleaning solution circulates through the processing equipment. The cleaning and rinsing solutions may circulate through the CIP system as substantially continuous streams, or as discrete slugs of solution separated by 25 pockets of air.

In one embodiment of the present invention, the cleaning step is performed without having first performed any kind of pre-rinse step. As commonly understood, a "pre-rinse" step is a procedure by which typically fresh water is circulated 30 through the handling or processing equipment at a temperature of between about 35° C. to about 40° C. in order to remove or loosen various soils so as to conserve detergent or improve the cleaning efficacy of the cleaning step. Typically, the volume of water used in the pre-rinse step is roughly the 35 same as the volume of cleaning solution and post-rinse solution that are circulated through the system during the cleaning and rinsing steps, respectively. However, generally, the volume of water used in the pre-rinse step is at least 75% of the volume of cleaning solution that is used during the 40 cleaning step.

In another embodiment of the present invention, a volume of cleaning solution is circulated through the handling or processing equipment in a plurality of passes to effect a reduction of the soils on the equipment surfaces. However, 45 after the first pass of the cleaning solution, a first portion of the cleaning solution is purged from the equipment. In certain embodiments this first portion constitutes the "first runnings" or the first slug of cleaning solution to pass through the equipment. As discussed above, certain CIP system contain significant quantities of food or beverage that, due to the system design, cannot be automatically drained from the system. This first portion of cleaning solution contacts the free-standing food or beverage remaining in the system prior to the cleaning step and "drives" it out 55 of the system. Accordingly, this first portion of cleaning solution is purged so as to not reduce the efficacy of the remaining detergent within the system. The remaining cleaning solution is continued to be passed through the equipment for the remainder of the cleaning step. In certain embodiments, the first portion of cleaning solution that is purged from the equipment comprises less than 25% by volume of the total volume of cleaning solution circulated during the first pass. In other embodiments, the purged portion comprises less than 15%, or less than 5% of the total volume of 65 cleaning solution circulated during the first pass. By purging the first slug of cleaning solution after the first pass, the need

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for a conventional pre-rinse step is eliminated thereby conserving considerable amounts of fresh water.

In another embodiment of the present invention, the cleaning step comprises introducing a first portion of a cleaning fluid, preferably fresh water, into the equipment thereby contacting the surfaces thereof. Subsequently, a second portion of cleaning fluid is introduced into the equipment thereby contacting the surfaces thereof. The second portion of cleaning fluid comprises an acidic detergent composition. The first and second portions of cleaning fluid are circulated simultaneously through the equipment for the duration of the cleaning step. Note, in this embodiment, the first portion of cleaning fluid is not purged from the system. In this embodiment, the first portion of cleaning fluid picks up and dilutes the free-standing quantities of food or beverage remaining in the system so as not to reduce the effectiveness of the detergent that is contained within the second portion of cleaning fluid. Again, the need for a pre-rinse step is eliminated thereby conserving water. In certain embodiments, the first portion of cleaning fluid comprises less than 25% by volume of the total cleaning fluid used in the cleaning step. In other embodiments, the first portion of cleaning fluid comprises less than 15%, or less than 5% by volume of the total cleaning fluid used in the cleaning step.

In yet another embodiment of the present invention, a pre-rinse step is performed prior to the cleaning step. However, the volume of pre-rinse fluid used is less than 50% of the volume of cleaning solution used in the cleaning step. In other embodiments, the volume of pre-rinse fluid used is less than 40%, preferably less than 25%, and most preferably less than 10% of the volume of cleaning solution used in the cleaning step. It is the primary function of the pre-rinse step to reduce the amount of "free-standing" food or beverage that cannot otherwise be drained from the system prior to the cleaning step. Therefore, it is not a target goal of the pre-rinse step to loosen or remove soils that are adhered to the surfaces of the equipment. Rather, the pre-rinse is primarily intended to reduce the amount of food or beverage to an acceptable level that does unacceptably interfere with or prevent the detergent used in the cleaning step from effecting the necessary system cleaning. Thus, the pre-rinse step may employ lower temperatures than conventional pre-rinse operations, thereby resulting in additional energy savings. For example, the pre-rinse solution or fluid may have a temperature of less than 35° C., less than 30° C., less than 25° C., between about 10° C. to about 35° C., or between about 15° C. to about 30° C.

It has been discovered that in order to obtain effective cleaning from the cleaning step, the food or beverage handling and processing equipment should contain less than 12% by volume of residual food or beverage, based upon the volume of cleaning solution to be circulated through the equipment, prior to the cleaning step, or at least prior to the introduction of detergent into the equipment during the cleaning step. In certain embodiments, the level of such food or beverage soils should be less than 10% by volume, or even less than 5% by volume, based upon the volume of cleaning solution to be circulated through the equipment.

After the specified time period, the surface is rinsed. In the rinsing step, the surface is contacted with a rinse solution for a sufficient time to remove any cleaning solution residue. Preferably, the rinse solution comprises fresh water (i.e., water that has yet to be cycled through the equipment). Preferably, the surface is rinsed for a specified period of from about 2 to about 20 minutes, and more preferably from about 4 to about 16 minutes, at a temperature of from about

5° C. to about 40° C., preferably from about 10° C. to about 35° C., and more preferably from about 15° C. to about 30° C. After the rinsing step, the surface is clean and descaled. Thus, in a single cleaning cycle the inventive method provides for the removal of at least about 90% of the food 5 and/or beverage soil on the equipment surface, preferably from about 90%-99.9% of the soil is removed, and more preferably from about 95-98%, based upon the initial amount of food and/or beverage soil on the equipment surface prior to the cleaning cycle.

The inventive method also preferably sanitizes the surface at cleaning temperatures of at least about 40° C., resulting in at least a 4-log reduction, and more preferably at least a 5-log reduction, in the amount of bacteria or microorganisms on the target surface after a single cleaning cycle. As used 15 herein, the term "cleaning cycle" refers to a single cleaning step, followed by a post-rinse step, and in certain embodiments, without a pre-rinse step. Thus, in certain embodiments, in a single cleaning cycle, a soiled surface is not pre-rinsed, but is first contacted with the cleaning solution 20 for a specified period of time, and is then rinsed with the rinsing solution to directly thereafter yield a surface that is cleaned, sanitized, and descaled.

In one embodiment, the cleaning solution is run through the equipment for a single cleaning cycle and then drained 25 from the equipment and discarded. That is, once the cleaning solution is drained after the single cleaning cycle, it is not reintroduced into the equipment during a subsequent cleaning cycle. Thus, in this embodiment, the cleaning solution is a single-use solution.

In another embodiment according to the invention, the rinse water is recovered after the rinsing step and reused during a subsequent cleaning cycle. Preferably, the rinse water is diverted to a holding tank after the rinsing step and is used in the cleaning solution of a subsequent cleaning 35 cycle. According to this embodiment, a quantity of the detergent composition is introduced into the recovered rinse solution to produce a cleaning solution for the subsequent cleaning cycle having the desired detergent concentration, as described herein.

The cleaning solution comprises a non-chlorine detergent composition that provides cleaning and descaling functionality, and is also capable of providing sanitizing under certain conditions. The preferred cleaning compositions for use in the cleaning solutions of the present invention are 45 described in U.S. Patent Application Publication 2006/ 0035808, incorporated by reference herein in its entirety. These detergent compositions comprise a fatty alkyl-1,3diaminopropane or salt thereof, and one or more inorganic and/or organic acids. In addition, the detergents may also include low-foaming non-ionic surfactants, as well as coupling agents. The compositions can also include one or more additional ingredients such as one or more sequesterants, builders, and chelating agents. It is also particularly preferable to include a quantity of a lower-alkyl sulfonic acid 55 (such as methanesulfonic acid) to further enhance the cleaning performance of the composition. A particularly preferred cleaning composition is commercially available under the name ZoneTM, from DeLaval Manufacturing. As mentioned above, it is preferred that the cleaning solution is discarded 60 after a single cleaning cycle; although the rinse water can be recovered and reused in the cleaning solution of a subsequent cleaning cycle. In an alternative embodiment, the cleaning solution can also be saved and re-used for other types of cleaning such as external surface cleaning of the 65 milking parlor. However, it is preferable that the cleaning solution not be re-used in the CIP system.

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The detergent concentrates to be used with the present cleaning methods preferably comprise from about 0.01-5% by weight of a fatty alkyl 1,3-diaminopropane or salt thereof, based upon the total weight of the composition taken as 100% by weight. The fatty alkyl-1,3-diaminopropanes for use in the cleaning composition have the general formula

R—NH—CH₂CH₂CH₂NH₂,

wherein R is a substituted or unsubstituted, straight or branch, saturated or unsaturated C4-C22 alkyl group in an acid matrix. It is preferable that the R group correspond as closely as possible to the fatty alkyl group distribution of the soil being cleaned. Preferably, the fatty alkyl-1,3-diamino-propane is derived from natural sources, such as coconut, soy, tallow, or oleo sources. Fatty alkyl-1,3-diaminopropanes can be used as amines or can be converted into diamine salts through a reaction with low alkyl carbon acids such as formic acid, acetic acid, or any other organic acids. Mono and diacetate salts of fatty alkyl-1,3-propylenediamines (alone or in combination) are particularly preferred. The mono and diacetate salts are prepared in situ by mixing of the amines with controlled amounts of acetic acid prior to adding any other ingredients.

Particularly preferred diaminopropane compositions are commercially available from Akzo Nobel under the name DUOMEEN. The DUOMEEN family includes Duomeen® C (Coco Alkyl), Duomeen® CD (Distilled Coco Alkyl), 30 Duomeen® S (Soya Alkyl), Duomeen® SV (Soya Alkyl vegetable derived), Duomeen® O (Oleo Alkyl), Duomeen® OL (Oleo Alkyl), Duomeen® T (Tallow Alkyl). These compositions are also available as diacetate salts, a neutralized product formed with acetic acid, such as Duomac® T (Tallow Alkyl diacetate salts) and Armohib® B-101. Additional diaminopropane compositions are available from Clariant under the name GENAMIN and includes Genamin® OLP 100 (Oleyl propylenediamine), Genamin® TAP 100 (Tallow Alkyl propylenediamine), Genamin® TAP 40 100 D (Tallow Alkyl propylenediamine, distilled), Genamin® LAP 100 (Lauryl propylenediamine). Yet additional diaminopropane compositions are available from Corsicana Technologies under the name CORSAMINE, such as Corsamine® DC (Coco Alkyl), Corsamine® DO (Oleo Alkyl), and Corsamine® DT (Tallow Alkyl). Other specific examples of fatty alkyl-1,3-propylenediamines are disclosed in U.S. Patent Application Publication 2006/0035808, and are incorporated by reference herein.

The detergent concentrates also preferably comprises from about 1-80% by weight acid (either organic or inorganic), more preferably from about 10-60% by weight, and even more preferably from about 15-50% by weight based on the total weight of the composition taken as 100% by weight. The acids for use in the composition can be any organic or inorganic acids known to those skilled in the art. Preferred organic acids include weak C1 to C4 carboxylic acids such as acetic acid, hydroxyacetic acid, propionic acid, hydroxypropionic acid, α-ketopropionic acid, citric acid, butyric acid, mandelic acid, valeric acid, succinic acid, tartaric acid, malic acid, oxalic acid, fumaric acid, adipic acid, or mixtures thereof. Other preferred organic acids include maleic acid, sorbic acid, benzoic acid, glutaric acid, adipic acid, α-hydroxy acids such as glycolic acid and lactic acid, ethylenediaminetetraacetic acid (EDTA), phosphonic acid, octyl phosphonic acid, acrylic acid, polyacrylic acid, aspartic acid, polyaspartic acid, p-hydroxybenzoic acids, and combinations thereof. Yet additional preferred organic

acids are those having the general formula R¹—SO₃H wherein R¹ is a C1-C16 alkyl group.

Preferred inorganic acids include mineral acids such as sulfuric acid, nitric acid, phosphoric acid, sulfamic acid, hydrochloric acid, and mixtures thereof. Methanesulfonic acids, sulfamic acids, and phosphoric acids are also helpful in descaling soiled surfaces.

Preferably, the inventive detergent compositions comprise water soluble acids in sufficient concentration to provide use solutions having a pH from about 0.1-5.5 preferably from about 1.0-3.5, more preferably from about 1.5-3.0, and most preferably from about 2.0-2.5. Exemplary water soluble acids include citric acid, phosphoric acid, methanesulfonic acid and sulfamic acid. Phosphoric acid is particularly advantageous acid because it also provides some hydrotropic properties to solubilize nonionic surfactants that may be incorporated with the detergents. Phosphoric acid, methanesulfonic acid, and sulfamic acid are also particularly advantageous for use in cleaning dairy pipelines as they tend to dissolve milk stone.

Surfactants are important ingredients in detergents because they impart beneficial properties to the detergents, such as wetting, lowering surface tension, and cleaning assistance. However, many surfactants tend to foam when 25 agitated. In CIP systems, because it is desirable to create as short a wash time as possible, excessive or long lasting foam is highly undesirable. CIP systems are particularly prone to foaming due to the agitation and slug action of the cleaning detergents. Also, protein soils, in general, naturally tend to 30 produce foam. Therefore, it is important in the context of these systems to select surfactants which are non-foaming or very low foaming for use in accordance with the claimed method. However, in applications where foaming is not a concern, such as the cleaning of utensil surfaces or storage 35 tanks, high foaming surfactants may be used.

A number of different surface active agents can be used in the present method and include anionic, nonionic, cationic, amphoteric, and zwitterionic surfactants, or mixtures thereof which are stable in highly acidic conditions. Specific 40 examples of such surfactants are described in detail in U.S. Patent Application Publication 2006/0035808 and are incorporated by reference herein. Preferably, detergent concentrate compositions for use according to the present method comprise from about 0-15% by weight of a surfactant, more 45 preferably from about 0.10-15% by weight, even more preferably from about 0.50-10% by weight, still more preferably from about 1.0-8% by weight, and most preferably, from about 2-6% by weight. Mixtures of two or more surface active agents are particularly preferred for the 50 claimed method.

Nonionic surfactants tend to lower the detergent surface tension, improve the wettability of the surface being cleaned, and solubilize the soils in the inventive detergents. Thus, these are particularly preferred for use in the claimed 55 method of cleaning CIP systems. Preferred nonionic surfactants include capped or uncapped poly-lower alkoxylated higher alcohols or ether derivatives thereof, in which the alcohol or ether contains 9 to 18 carbon atoms and the number of moles of lower alkylene oxide (2 or 3 carbon 60 atoms) is from 3 to 12. Exemplary alkyl alkoxylated alcohols are available from BASF under the name PLURAFAC (Fatty alcohol alkoxylates) such as, Plurafac® LF-303 (polyglycol ether), Plurafac® LF-305 (C8-C14 alkyl chain), Plurafac® S-305LF, Plurafac® SLF-18B (C6-C10 ethoxy- 65 lated linear alcohol), Plurafac® SLF-18B45, and Plurafac® LF-4030. Another preferred nonionic surfactant is available

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from Clariant under the name GENAPOL®, such as GENAPOL® EP 0244 (Alkyl alkoxylate).

Even more preferably, the claimed method involves a dual surfactant system using two different nonionic surfactants, which surprisingly, was found to result in less foaming in the CIP system when compared with cleaning methods including single surfactant systems.

It is also preferred that the compositions include the lower alkanesulfonic acid, methanesulfonic acid, CH₃SO₃H, at a 10 level of about 0-40% by weight of a lower alkyl sulfonic acid, more preferably from about 1-30% by weight, even more preferably from about 2-25% by weight, and most preferably from about 5-20% by weight, based upon the total weight of the composition taken as 100% by weight. Methanesulfonic acid is a strong organic acid (pKa=-1.9) distinguished by a particularly high capacity for solvating numerous heavy metals. It was discovered that the addition of methanesulfonic acid to the detergent formulations used in the claimed method greatly improved the cleaning performance of the detergent, especially in removing protein films. Methanesulfonic acid and its metal salts are highly soluble in water, and less corrosive than other strong inorganic acids. Methanesulfonic acid is biodegradable and recyclable. Methanesulfonic acid is generally less toxic than fluoroboric acid and fluorosilicic acid. Other lower alkyl (C_1-C_{16}) carbon chain sulfonic acids may be used in the claimed method, such as ethanesulfonic acid, propanesulfonic acid, and butanesulfonic acid.

As noted above, the claimed method preferably provides cleaning and descaling functionality in a single cleaning step with a single cleaning product, which is also capable of providing sanitizing under certain conditions. Thus, it is desirable for the compositions used in the claimed method to include antibacterial and sanitizing agents. Specific examples of antibacterial and sanitizing agents that can be included in compositions used with the claimed method are provided in U.S. Patent Application Publication 2006/ 0035808, and are incorporated by reference herein. However, it is preferred that the compositions used in the present method are substantially free of any peroxides, such as hydrogen peroxide. As used herein the term "substantially free," means that the composition contains less than about 0.01% by weight of peroxide. Even more preferably, the compositions contain no peroxides.

Particularly preferred coupling agents for use in the claimed method include nontoxic biodegradable monohydric alcohols, selected polyhydric alcohols, aromatic alcohols, and aliphatic alcohols. Preferred monohydric alcohols are selected from the group consisting of isopropyl, methyl, ethyl, propyl, isopropyl, n-butyl, isobutyl, tert-butyl, benzyl, and allyl alcohols, and mixtures thereof. Preferred polyhydric alcohols are selected from the group consisting of propylene glycol, 1,3-propanediol, 1,2-butanediol, polyethylene glycol 400, glycerol, and 1,4-butanediol, and mixtures thereof. It is particularly preferred to use a monohydric alcohol in combination with a polyhydric alcohol in the cleaning compositions of the claimed method. These agents are preferably included in the compositions used with the present method at a level of up to about 20% by weight coupling agent, more preferably from about 0.5-10% by weight, even more preferably from about 1-8% by weight, and most preferably from about 1.5-6% by weight, based upon the total weight of the composition taken as 100% by weight.

Finally, compositions used in the claimed method can include sequestrants, builders, and chelating agents to soften or treat water and to prevent the formation of precipitates or

other salts in the CIP system. Generally, sequestrants complex or coordinate the metal ions commonly found in the service water and thereby prevent the metal ions from interfering with the functioning of the detersive components within the composition. Preferred examples of these optional ingredients are disclosed in U.S. Patent Application Publication 2006/0035808, and are incorporated by reference herein.

The detergent concentrate is capable of being diluted with water to form a use solution (i.e., the cleaning solution).
Preferably, the concentrate is diluted at a weight ratio of between is diluted at a weight ratio of between about 1:10 to 1:400, more preferably between about 1:50 to 1:300, and most preferably between about 1:100 to 1:250. The dilution ratio, when expressed as a percentage of the volume of detergent concentrate per total volume of solution, may be from about 0.2 to about 0.8%, preferably from about 0.3 to about 0.6%, more preferably from about 0.4 to about 0.5%, and most preferably about 0.4%. The pH of the diluted use solution is preferably less than about 5.5, preferably between about 0.1-5.5, more preferably between about 1.0-3.5, even more preferably between about 1.5-3.0, and most preferably between about 2.0-2.5.

EXAMPLES

The following examples set forth preferred cleaning methods in accordance with the invention. It is to be understood, however, that these examples are provided by way of illustration and nothing therein should be taken as a 30 limitation upon the overall scope of the invention.

Example 1

Cleaning Performance of Single Cycle Method

The cleaning efficacy of the claimed method was tested. First, soiled panels were prepared according to the following procedure. Sixty-three stainless steel, plastic, or glass panels measuring $3"\times6"\times0.0037"$, having a ½ inch hole at one end 40 were at first washed with a powder chloro-alkaline detergent, rinsed with water and wiped with xylene, then with isopropanol, followed by drying in an oven (100-110° C., for 10-15 minutes) to insure complete evaporation of the solvents. The panels were suspended in the oven by attaching 45 a rigid wire hanger to the panel hole, so that no contact was made with the oven or other items within the oven. The dried panels were then removed from the oven, and allowed to cool for at least 20 minutes. The panels were then carefully handled so as to eliminate contact with soil sources, and the 50 initial weight of each panel was recorded to the nearest 0.1 mg.

Three 12 fl. oz. (354 mL) cans of Nestle Carnation evaporated milk were emptied into to a 1 L beaker, along with one 12 fl. oz. (354 mL) can of de-ionized water to make 55 a 75% solution of Carnation Milk as the milk soil. The mixtures were stirred to insure homogeneity. The panels were placed in the milk by setting the end without the hole on the bottom of the beaker and propping the other end of the panel against the side of the beaker. Approximately 1/8 of 60 the panel was immersed in the milk. The panels were allowed to sit in the milk for 15 minutes and then drained in the air for 5 minutes. Each panel side was then rinsed with 50 ml of 400 ppm of synthetic hard water previously heated to 90-100° F. Care was taken to pour the rinse water over 65 each side of the panel so as to contact all of the soiled areas of the panel. The rinse water was allowed to drain off each

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panel and then the panels were hung in a 40° C. oven to dry. The panels were then removed from the oven and allowed to cool for at least 15 minutes. After cooling, the panels were weighed and each weight was recorded to the nearest 0.1 mg. The soil deposition, rinsing, drying and weighing cycle was carried out a total of five times for each panel, or until the soil weight fell within the range of about 18-30 mg.

The soiled panels were then washed in a 1 L beaker using a cleaning solution at varying temperatures without prerinsing the panels with water. The cleaning solution was prepared by mixing the ingredients as shown in Table 1 below, and diluting to a 0.4% use concentration.

TABLE 1

Ingredient	Percentage $(w/w)^1$	Use Concentration 0.4% (v/v) Percentage (w/w) ¹
Water	60.50	0.02735
Acetic Acid	0.25	0.0011
Fatty alkyl 1,3-diaminopro	pane ² 0.25	0.0011
5 Genapol EP 0244 ³	2.00	0.0090
Plurafac SLF 18B-45 ⁴ (100	0%) 1.00	0.0045
Phosphoric acid, 75%	15.00	0.0678
Methanesulfonic acid, 70%	6 15.00	0.0678
Isopropyl alcohol, 99%	3.00	0.0136
Propylene glycol, USP	3.00	0.0136
Acid Red 27 ⁵	0.0025	~0.0000
TOTAL	100.00	0.4520

¹Based upon the total weight of the composition taken as 100% by weight.

To test additional organic soil load, Carnation Evaporated Milk was added to the heated cleaning solution at 1%, 2%, 3%, 4%, 5%, 8%, and 10% milk solutions, respectively, and allowed to mix for 2 minutes. For each concentration of milk, the cleaning solution was heated to three different temperatures, 40° C., 50° C., and 60° C., respectively, using a hot plate. The pH of the cleaning solution at each temperature was recorded. For each temperature three different test panels were used. Thus, a total of nine panels were tested for each milk solution.

Each test panel was first immersed in the cleaning solution for a period of 8 minutes with agitation via a magnetic stir bar, while the designated temperature was maintained with a hot plate. After the wash, each panel was removed from the wash bath and immediately rinsed in tap water for about 5 seconds. The panel was then suspended within an oven at about 40° C. for a period of about 15 minutes to dry. The panel was removed from the oven, cooled in the air for about 30 minutes and then reweighed. The weight of the panel after the wash cycle was then compared with the soiled weight thereof before the wash cycle to determine the percent soil removed. The results are provided in Table 2 below.

²Genamin OLP 100 (available from Clariant) or Duomeen SV (available from Akzo Nobel)

³Low-foaming nonionic surfactant available from Clariant

⁴Nonionic surfactant available from BASF

⁵Acid stable dye

TABLE 2

Panel #	Temperature/ pH	Initial Weight (g)	Soiled Weight (g)	Total Amount of Soil (g)	Weight After Cleaning (g)	Weight of Soil Removed (g)	% Soil Removed	Average Cleaning %
	P	(6)		Cleaning Soluti				0144444
			0.470 080	Cleaning Solut	IOII/ I /O IVIIIK III	Solution		
1	60° C./2.33	81.9718	81.9935	0.0217	81.9727	0.0208	95.85	97.52
2		82.1387	82.1643	0.0256	82.139	0.0253	98.83	
3	500 60 /2 22	82.7153	82.7388	0.0235	82.7158	0.0230	97.87	07.65
4	50° C./2.32	80.9883	81.0115	0.0232	80.9889	0.0226	97.41	97.65
5 6		81.0394 80.8349	81.0656 80.862	0.0262 0.0271	81.0396 80.8359	0.0260 0.0261	99.24 96.31	
7	40° C./2.31	80.7595		0.0271	80.7599	0.0236	98.33	96.86
8	10 0.,2.51		81.2102	0.0189	81.1917	0.0185	97.88	70.00
9			82.2687	0.0231	82.2469	0.0218	94.37	
			0.4% Use	Cleaning Soluti	ion/2% Milk in	Solution		
10	60° C./2.36	81.7295	81.7592	0.0297	81.73	0.0292	98.32	98.25
11			81.7184	0.0214	81.6972	0.0212	99.07	3 0.20
12		81.9070	81.9297	0.0227	81.9076	0.0221	97.36	
13	50° C./2.35	82.8677	82.8921	0.0244	82.8683	0.0238	97.54	96.42
14		80.2189	80.2413	0.0224	80.2196	0.0217	96.88	
15		82.7143	82.7356	0.0213	82.7154	0.0202	94.84	
16	40° C./2.33		82.7063	0.0205	82.6877	0.0186	90.73	94.28
17			82.4661	0.0280	82.4387	0.0274	97.86	
18		82.4823	82.5067 0.4% Use	0.0244 Cleaning Soluti	82.4837 ion/3% Milk in	0.0230 Solution	94.26	
19	59° C./2.36		82.3708	0.0241	82.3477	0.0231	95.85	96.93
20			82.2352	0.0228	82.2129	0.0223	97.81	
21	500 0 /2 26		81.4382	0.0243	81.4146	0.0236	97.12	07.25
22 23	50° C./2.36	80.3738	80.6034	0.0276 0.0246	80.5762 81.8762	0.0272 0.0238	98.55 96.75	97.35
24			82.9033	0.0246	82.8795	0.0238	96.75	
25	40° C.	80.0772		0.0309	80.0814	0.0256	86.41	87.48
26	10 0.		82.3758	0.0246	82.3544	0.0214	86.99	07.10
27			80.4873	0.0283	80.4621	0.0252	89.05	
			0.4% Use	Cleaning Soluti	ion/4% Milk in	Solution		
28	60° C.	80.5393	80.5667	0.0274	80.5402	0.0265	96.72	91.02
29			80.7458	0.0185	80.7296	0.0162	87.57	3 210 2
30		82.2801	82.3095	0.0294	82.2834	0.0261	88.78	
31	51° C.	80.2476	80.2725	0.0249	80.2497	0.0228	91.57	91.40
32		80.1684	80.1879	0.0195	80.1704	0.0175	89.74	
33		82.6031	82.634	0.0309	82.6053	0.0287	92.88	
34	41° C.		80.6209	0.0324	80.5935	0.0274	84.57	85.55
35		80.3454		0.0296	80.3495	0.0255	86.15	
36		80.7729		0.0256 Cleaning Soluti	80.7765 ion/5% Milk in	0.0220 Solution	85.94	
			0.470 080	Cicaming Solut.	IOII/ J /U IVIIIK III	Solution		
37	60° C.	81.3186	81.3438	0.0252	81.3228	0.0210	83.33	89.85
38		80.6691	80.699	0.0299	80.6717	0.0273	91.30	
39		80.5116	80.5391	0.0275	80.513	0.0261	94.91	
40	51° C.	80.1872	80.2125	0.0253	80.1904	0.0221	87.35	85.51
41		82.0546	82.0837	0.0291	82.0585	0.0252	86.60	
42		80.1004	80.1308	0.0304	80.1057	0.0251	82.57	
43	40° C.		82.8604	0.0258	82.84	0.0204	79.07	78.75
44		82.2038		0.0262	82.2098	0.0202	77.10	
45		80.8186	80.8482	0.0296 Cleaning Soluti	80.8245	0.0237	80.07	
			U.T/U USC	Cicaming Bolut	CIPO O IVIIIK III	Solution		
46	60° C.		83.1014	0.0290	83.0756	0.0258	88.97	87.17
47		82.0111	82.0355	0.0244	82.0143	0.0212	86.89	
48		81.9869	82.0092	0.0223	81.9901	0.0191	85.65	
49	50° C.		82.8322	0.0236	82.8122	0.0200	84.75	85.01
50			82.1521	0.0238	82.1315	0.0206	86.55	
51			82.9887	0.0246	82.9681	0.0206	83.74	<u> </u>
52	40° C.		82.6257	0.0273	82.6023	0.0234	85.71	81.06
53		81.6834		0.0207	81.6882	0.0159	76.81	
54			82.5083 0.4% Use	0.0212 Cleaning Solution	82.4912 on/10% Milk in	0.0171 Solution	80.66	
			∪.+/0 USE	Creaming Somme	OIV I O /O IVIIIK III	DOIUHOH		
		927672	82.7895	0.0223	82.7718	0.0177	79.37	84.07
55	60° C.	82.7072						
55 56	60° C.		81.4885	0.0296	81.4631	0.0254	85.81	
	60° C.	81.4589		0.0296 0.0285	81.4631 79.437	0.0254 0.0248	85.81 87.02	
56	60° C.	81.4589 79.4333	81.4885					82.84
56 57		81.4589 79.4333 80.2776	81.4885 79.4618	0.0285	79.437	0.0248	87.02	82.84

TABLE 2-continued

Panel #	Temperature/ pH	Initial Weight (g)	Soiled Weight (g)	Total Amount of Soil (g)	Weight After Cleaning (g)	Weight of Soil Removed (g)	% Soil Removed	Average Cleaning %
61 62 63	40° C.	82.5880 82.7265 81.6425		0.0213 0.0276 0.0296	82.5936 82.7298 81.6473	0.0157 0.0243 0.0248	73.71 88.04 83.78	81.85

Example 2

Additional Acidic Detergent Formulations for Single Cycle Method

In this Example, the amount and type of ingredients in the acidic detergent formulations were varied and tested for cleaning performance. The formulations were also evaluated for stability over a 14 day period, when stored at 40° C. and 20 45° C., respectively. The formulations are provided in Table 3 below, along with the corresponding cleaning performance. To test the average % cleaning, each formulation was diluted to a 0.25% (v/v) use concentration and was tested at 60° C. All of the use solutions showed stability over a period 25 of 14 days.

TABLE 3

		11 110				
Ingredients	Formulation					
$(\mathbf{w}/\mathbf{w})^1$	\mathbf{A}	В	С	D	Ε	
Water	48.51	48.00	60.50	8.35	10.00	
Acetic acid 100%	0.33	0.50	0.25	0.33	0.50	
Fatty alkyl 1,3-diamino- propane ²	0.33	0.50	0.25	0.33	0.50	
Genapol EP 0244	2.60	2.50	2.00			
Plurafac18B- 45	1.44	1.50	1.00	1.95	1.50	
Phosphoric acid 75%	19.50	25.00	15.00	45.50	45.00	
Methanesul- fonic Acid	19.50	15.00	15.00			
Isopropanol	3.90	3.50	3.00			
Propylene Glycol	3.90	3.50	3.00	3.90	3.00	
Plurafac LF303				1.95	2.50	
Citric Acid (Anhydrous)				0.00	0.00	
Sodium Xy- lene Sulpho- nate 40%				32.50	30.00	
Emery 658				1.30	2.00	
Glycolic				3.90	5.00	
Acid 70						
Plurafac S-305LF						
Total Average	100.00 81%	100.00 79%	100.00 83%	100.00 83%	100.00 84 %	
Cleaning %						

¹Percentage by weight, based upon the total weight of the composition taken as 100% by weight.

Example 3

Comparison of Single Cycle Method Using Chlorinated Alkaline Detergent

In this Example, the cleaning efficacy of the claimed method using the cleaning solution from Table 1 was compared to a commercially available liquid chloro alkaline detergent sold under the name Dynamate II, available from DeLaval Manufacturing. The same procedures outlined in Example 1 were followed using a 0.4% use solution of liquid Dynamite II detergent to clean milk soiled panels. A control was also prepared and cleaning efficacy was evaluated without any additional milk soil in the cleaning solution. The cleaning performance of the detergent was then tested using from 1-10% additional milk soil loads in the cleaning solutions as described in Example 1 above. The results for the commercially available chloro alkaline detergent versus the cleaning solution from Example 1 are summarized in Table 4 below.

TABLE 4

Use concentration/milk concentration	Wash Temper- ature (° C.)	Table 1 Solution Average % of Soil Removed	Dynamate II Solution Average % of Soil Removed
Detergent 0.4% Solution/	60	95	99
No Additional Milk Soil	50	91	99
	40	86	97
Detergent 0.4% Solution/	60	98	67
1% Additional Milk Soil	50	98	87
	40	97	84
Detergent 0.4% Solution/	60	98	67
2% Additional Milk Soil	50	96	71
	40	94	64
Detergent 0.4% Solution/	59	97	71
3% Additional Milk Soil	50	97	67
	40	88	67
Detergent 0.4% Solution/	60	91	64
4% Additional Milk Soil	51	91	63
	41	86	58
Detergent 0.4% Solution/	60	90	57
5% Additional Milk Soil	51	86	60
	40	79	55
Detergent 0.4% Solution/	60	87	60
8% Additional Milk Soil	50	85	61
	40	81	52
Detergent 0.4% Solution/	60	84	63
10% Additional Milk Soil	50	83	67
	40	82	64

As can be seen from Table 4 above, there was a substantial decrease in the cleaning performance of the commercially-available chloro alkaline detergent in the single cycle cleaning method as the load of additional milk soil is increased in the cleaning solution. A 30-35% performance loss was observed in the Dynamate II solution compared to the cleaning solution from Table 1. In addition, once the soil load reached 4%, the cleaning performance of Dynamate II remained virtually the same, removing only about 60% of

²Genamin OLP 100 (available from Clariant) or Duomeen SV (available from Akzo Nobel)

the soil load. This was believed to be due to the higher levels of residual milk soil (that are normally removed via the pre-rinse) eventually depleting the chlorine in the Dynamate II solution, leaving only the alkaline cleaner to clean the remaining milk soil. This decreased performance was not 5 seen in the cleaning solution from Table 1. For example, when looking at the 10% milk soil load, there was a 34-37% decrease in average cleaning performance compared to the no soil load for the Dynamate II solution, while the cleaning solution from Table 1 only experienced a 4-10% decrease 10 under identical wash conditions.

We claim:

- 1. A method of cleaning soiled food or beverage manufacturing and handling equipment, said equipment being a Clean-In-Place system, comprising:
 - a cleaning step in which the surfaces of said equipment are contacted with a volume of a cleaning solution comprising a non-chlorine detergent composition, wherein said volume of cleaning solution is circulated through said equipment for a period of time to effect a ²⁰ reduction of the soils on said equipment surfaces;
 - a post-rinsing step in which said equipment surfaces are rinsed with a rinse solution thereby removing residues of said detergent composition remaining on said equipment; and
 - a pre-rinse step in which a volume of pre-rinse fluid is circulated through said equipment prior to said cleaning step wherein the volume of pre-rinse fluid used is less than 50% of the volume of cleaning solution used in said cleaning step.
- 2. The method of claim 1, wherein the volume of pre-rinse fluid used in said pre-rinse step is less than 25% of the volume of the cleaning solution used in said cleaning step.
- 3. The method of claim 1, wherein the volume of pre-rinse fluid used in said pre-rinse step is less than 10% of the ³⁵ volume of the cleaning solution used in said cleaning step.
- 4. The method of claim 1, wherein said pre-rinse step reduces the amount of soils present in said equipment to less than 12% by volume based upon the volume of the cleaning solution used in said cleaning step.

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- 5. The method of claim 1, wherein said cleaning solution is substantially free of peroxides.
- 6. The method of claim 1, wherein said rinse solution comprises fresh water.
- 7. The method of claim 1, wherein said cleaning step comprises a single cleaning cycle wherein said cleaning solution is run through said equipment, drained from said equipment, and then discarded.
- 8. The method of claim 1, wherein said cleaning solution, once drained from said equipment, is not reintroduced into said equipment during a subsequent cleaning cycle.
- 9. The method of claim 1, said cleaning solution having a temperature of between about 25° C. to about 85° C. during said cleaning step.
- 10. The method of claim 1, said equipment surfaces being contacted with said cleaning solution for a time period of from about 2 to about 20 minutes.
- 11. The method of claim 1, wherein said method results in at least a 90% reduction in the soils present on said equipment surfaces, or at least a 4-log reduction in the level of bacteria present on said equipment surfaces.
- 12. The method of claim 1, wherein said rinse solution is recovered after said post-rinsing step and reused in the cleaning solution of a subsequent cleaning step.
- 13. The method of claim 12, wherein a quantity of said detergent composition is introduced into said recovered rinse solution in order to produce a cleaning solution having a detergent concentration of between about 0.2% to about 0.8% by volume.
- 14. The method of claim 1, said cleaning solution having a pH of between about 0.1 to about 5.5.
- 15. The method of claim 1, said equipment comprising milk- and beverage-handling equipment.
- 16. The method of claim 1, wherein said non-chlorine detergent composition comprises an organic acid, an inorganic acid, or a combination thereof.
- 17. The method of claim 1, wherein said non-chlorine detergent composition comprises a low-foaming non-ionic surfactant.

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